METHODS ABOUT THE CONTROL OF HYDRATION HEAT AND HYDRATION TEMPERATURE OF CEMENT

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ABSTRACT

This study aims to establish the methods to use to control the heat and temperature during the hydration of Portland cement. The importance of these control methods is to manage thermal distribution in the hydrating cement samples to eliminate the development of cracks. Ordinary Portland cement used during this study. Samples of 100 grams of cement were mixed with 10 % and 20% silica fumes, 50% and 70% fly ash as well as 40% of heavy water. Water to cement ratio of 0.4 was maintained for cement mixtures with silica fumes and fly ash. The experiments conducted showed that the concentration of silica fumes and fly ash have an inverse relation to the rate of heat change during the hydration process. The effect of heavy water on hydration process was also studied. 40% of heavy water was mixed with 100 grams of cement. Heavy water had a reduced rate of hydration, with a second peak of heat released of 1.3mW/g after 42 hours compared to the control experiment which had a second peak of heat released of 2.7mW/g after 18 hours.

KEYWORDS: Heat of Hydration, Admixtures, Exothermic Reaction, Isothermal Calorimetry & Filler Material

INTRODUCTION

The hydration of cement denotes the chemical reactions that occur when the cement is mixed with water. This process is an exothermic reaction, which involves the gradual emission of heat energy into the ambient until it sets. During the process of cement hydration, the internal structure will reach higher temperatures than on the surface. If the differences in temperature are high, then cracks may be formed in the structure (Gorzela, et al., 2016). The setting of cement means stiffening of the cement paste without significant development of compressive strength and typically occurs within a few hours. The cement used for this study is ordinary Portland cement. (Ylmen, 2013) showed that the four main mineral phases of cement are:

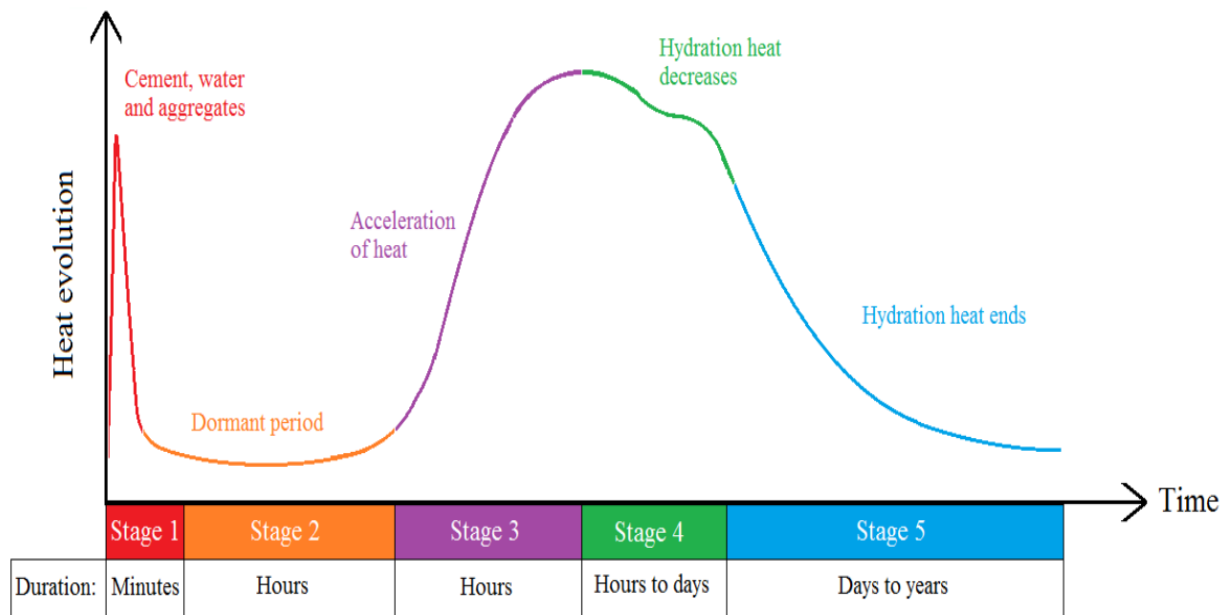Tetra-calcium alumino-ferrite $\quad Ca_4Al_2Fe_2O_{10}$(5-15 weight%)

Tri-calcium aluminate $\quad Ca_3Al_2O_6$ (5-10 weight%)

Di-calcium silicate (Belite) $\quad Ca_2SiO_4$ (15-30 weight%)

Tri-calcium silicate (Alite) $\quad Ca_3SiO_5$ (50-70%)

These four elements have been shown to assimilate for more than 90% of Portland cement (Zhi, 2005). Cements with higher content of tri-calcium silicate or tri-calcium aluminate have been shown by (Abla, 2013) to have higher heat generation. When the above fundamental compounds are mixed with water, alkali sulfates dissolve rapidly and release K[+], Na[+], and SO_4^{2-} ions into the solution. (Kim, 2010). Alite is estimated to hydrate about 5-25% by mass during its first stage of hydration. (Ivan, 1998) showed that in this initial stage of the hydration process, a very small amount of belite would react and in the process contribute to the Ca[2+] and OH[-] release in the liquid
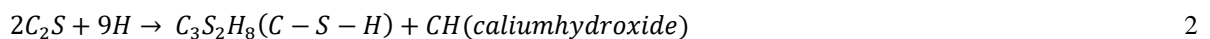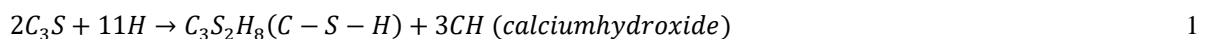
phase. (Karen & Nonat, 2011) demonstrated that the use of admixtures as a retarder reduces the reaction between alite and water. The dormant period as demonstrated by (Garrault, et al., 2001), shows a slow reaction of Alite. He proposed that the hydroxylated Alite formed during the initial stage has a lower solubility which is immediately proceeded by the acceleration stage. The rate of hydration in the acceleration stage is controlled by the formation of hydration products (Thomas, et al., 2009). Admixtures such as free lime added to Alite have been shown by (Juilland & Patrick, 2010) to prolong the induction period while higher amounts may shorten it. The hydration phases are indicated on Figure 1 below.

Figure 1: Stages of the Hydration Process (Kim, 2010).

Potential temperature rises or falls on cement hydration rate is assessed through the measurement of the heat of hydration. Temperature rise during mixing of cement and water is inherent as the interaction of anhydrous cement and water is exothermic (Ivan, 1998). These temperature changes and differences between the surface and the core of the concrete cause internal restraints. These restraints have been shown to be the cause of cracks which may be permanent if they occur during the contraction stage. However, if they occur during the expansion phase, they usually close due to the self-healing properties of concrete (Blomdahl, et al., 2015). The use of admixtures such as silica fumes and fly ash have been shown to decrease the strains during hydration (Institute, 2016).

When alite, C_3S, and belite, C_2S hydrates, calcium hydroxide (CH) and calcium silicate hydrate are formed. The hydration process can be written in the form of equation 1 and 2 below. The calcium to silicon ratio of the C-S-H gel varies but is normally in the range of 1.5-2.2 (Taylor, 1992).

$$2C_3S + 11H \rightarrow C_3S_2H_8 (C-S-H) + 3CH \ (calciumhydroxide) \qquad 1$$

$$2C_2S + 9H \rightarrow C_3S_2H_8 (C-S-H) + CH (caliumhydroxide) \qquad 2$$

According to, (Zainab, 2017), the amount of heat that the cement mix can be calculated using equations 3 to 5.

$$H_1 = \left(\frac{RC}{W_i}\right) - 0.8(T - t_d) \qquad 3$$

Where:

H_1 = heat of solution of dry cement, $kg/°C$

R = Corrected temperature, °C

C= heat capacity, $kJ/°C$

W_i = mass of sample on ignited basis, g

T= room temperature, °C

t_d=final calorimeter temperature, °C

$$H_2 = \left(\frac{RC}{W_i}\right) - 1.7(T - t_h) - 1.3(t_d - t_h) \qquad\qquad 4$$

Where:

H_2 = heat of solution of partially hydrated sample, kJ/kg

t_h= final calorimeter temperature at end

The heat of hydration of cement largely influenced by the reactions of tri-calcium aluminate and tri-calcium silicate (Steven, 2006) is hence calculated as, H:

$$H = H_1 - H_2 - 0.4(t_h - 25) \qquad\qquad 5$$

Chemical composition of ordinary Portland cement used in this study have the following compositions shown in Table 1 below.
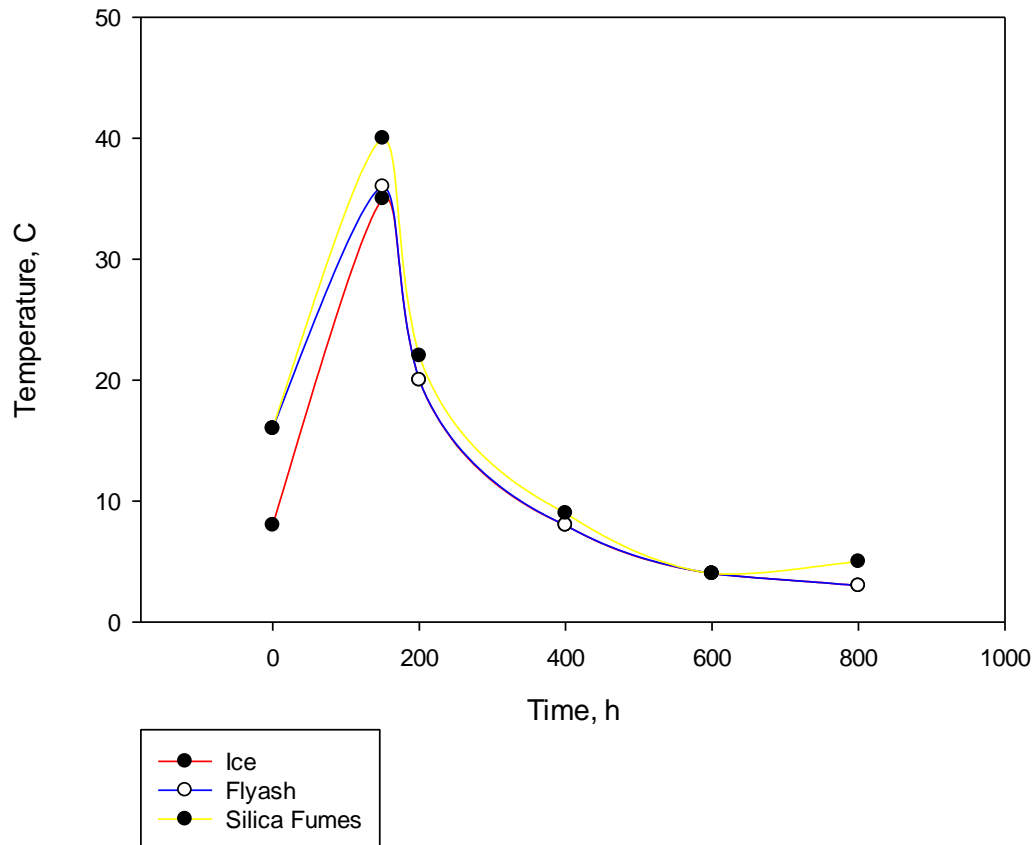
Table 1: Chemical Composition of Cement

Compound Composition	Chemical Composition	Percentage by Weight
Lime	CaO	62
Silica	SiO_2	19.66
Alumina	Al_2O_3	5.48
Iron Oxide	Fe_2O_3	3.72
Magnesia	MgO	2.87
Sulfate	SO_3	2.2
Free Lime	CaO	

METHODS USED TO CONTROL THE HYDRATION HEAT

The heat of hydration of Portland cement is influenced by various parameters namely, cement concentration, fineness, temperature, and water to cement ratio (Abbasb & Maida, 2017). However, the concentration of cement during the hydration process is the main component affecting the heat developed. Hence the hydration heat can be controlled by altering the amount of cement in the mix. (Poppe & Schutter, 2005) demonstrated that the hydration heat can be controlled through the introduction of binders. They performed an isothermal hydration test to evaluate the heat generated with different types of filler binders. Fillers being materials that will not ordinarily react with water during the hydration process contribute to being the most used binders and include, slag, fly ashes, silica fumes and limestone. (Sundblom, 2004)

Demonstrated that some fillers such as fly ash, silica fume and slag require an activator such as calcium oxide to react with water. The temperature gradient of the most used filler materials is as shown in Figure 2 below.

Figure 2: Temperature Gradient for Various filler Material (Sundblom, 2004).

In an experiment to determine the calorimetric behaviour as a result of altering the concentration of the filler material in the cement, the following experiments were conducted.

Silica Fumes

Silica fumes are reactive filler materials that have been shown by (Langan, et al., 2002) that their effect on hydration depends on the water to cement ratio. In an experiment conducted by (Ward & Yogendran, 1991) they showed that the hydration reaction of cement is altered due to the presence of silica fumes. Their results indicated that a higher mass ratio of silica fumes accelerates the hydration of cement. Silica fumes have been used largely as admixtures to cement paste as they have shown considerable impact in reducing thermal cracking during the cement hydration process (Bubshait, et al., 2007). In this paper, the effect of silica fume concentration on the hydration process is studied in experiment 1 below. The experiment is set up using isothermal calorimetry for thermal measurements, an approach for thermal measurements that was demonstrated to be accurate by (Sedaghat & Sandberg, 2013).

Experiment 1 Setup

The hydration test was performed on three 100-gram samples of ordinary Portland cement. Two of the cement samples were mixed with 10 grams (10%) and 20 grams (20%) by the mass ratio of silica fumes respectively, whilst the third sample was

without any filler to act as control as is shown in Table 2 below.

Table 2: Material Mix Components for Experiment 1

Material Mix	Mass (grams)		
	Cement	Silica Fumes	Water
Mix 1 (Test)	100	0	40
Mix 2	100	10	40
Mix 3	100	20	40

Apparatus

Insulated container- The container was used as a water bath. The insulation on the container was to reduce heat transfer between the ambient and the water bath.

Thermometer- Four NTC temperature probes were used during the experiment. Three of the probes were used to measure the temperature of the three samples whilst the fourth sensor was used as a reference sensor to measure the temperature of the water bath. The fourth temperature sensor was also used to control the water bath heater to maintain the experimental temperature of 23 ± 2°C.

Stirring assemble- The motorised stirring assemble was used to induce convectional currents on the water bath in order to improve heat distribution on the water bath.
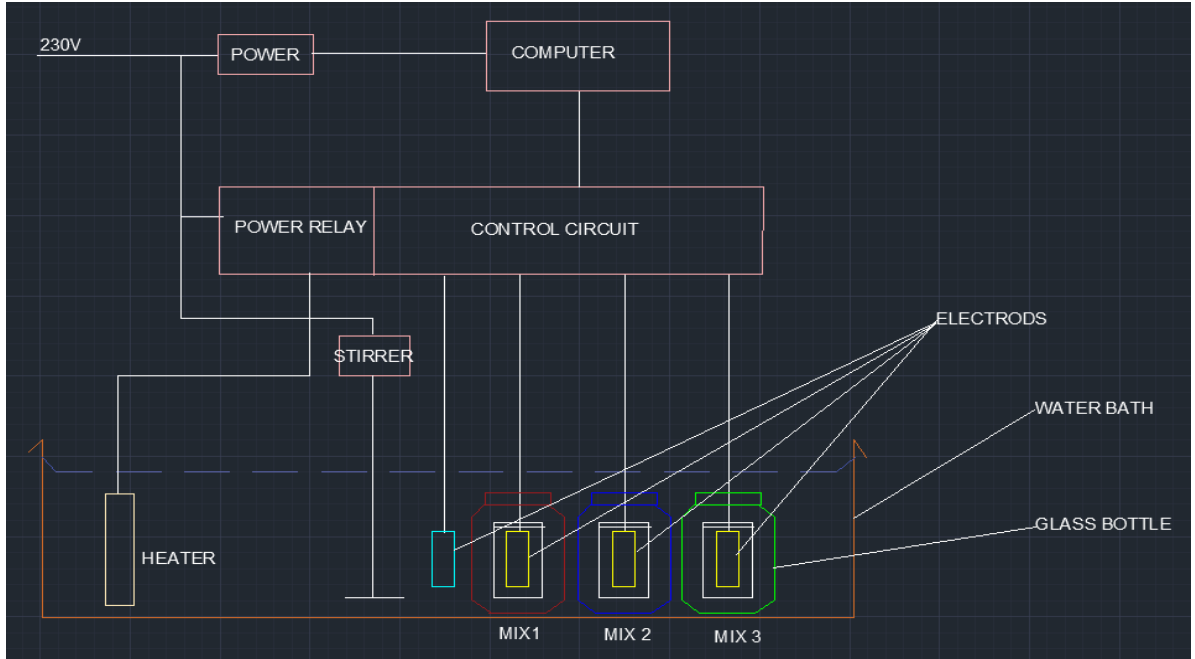
Analytical balance- An analytical balance was used to measure the mass of the samples to be used for the experiments.

Glass bottles- The glass bottles were used as containers for the prepared samples.

Mixer- A standard mixer with speed selection was used to mix cement with water and admixtures being tested to a uniform paste.

Controllers- The controller used for this experiment was a raspberry pi 4.0 model B.

The set up was maintained in a sealed and vacuumed glass bottle inside a water bath with a constant water temperature of 23 ± 2 °C. An equal amount of water of 120 ml was added to the three samples. The samples were then vigorously stirred for 8 minutes and then left to set. Differential electrodes were used to measure the rate of heat change during the hydration process of each mix as shown in Figure 3 below.

Figure 3: Experiment 1 Calorimeter Setup.

Calculating the Heat Capacity

The apparatus was set up as shown in Figure 3 above, the stirrer was switched on and the temperature of the water bath set to 23 °C. Observations of the calorimeter temperature were recorded in 10-minute periods. After 30 minutes when the water temperature had become uniform, the three different cement mixes were introduced in the water bath inside glass bottles.

After the introduction of the samples, temperature of the calorimeter was recorded at every 10-minute interval. The second 10-minute interval is the correction period, which was used to determine the actual heat of the cement mix samples.

$$R_0 = \alpha_{10} - \alpha_0 \qquad\qquad 6$$

$$R = R_0 - (\alpha_{20} - \alpha_{10}) \qquad\qquad 7$$

Where:

R_0= observed temperature rise, °C

R= corrected temperature rise, °C

α_0= temperature of the calorimeter when the samples were introduced, °C

α_{10}= temperature of the calorimeter at the end of the initial solution period, °C

α_{20}= temperature of the calorimeter at the end of the correction period, °C

The heat capacity of the calorimeter and the cement mix samples, C, was calculated as follows:

$$C = \frac{W_i[1072 + 0.4(30-t) + 0.5(T-t)]}{R} \qquad\qquad 8$$

Where:

C= heat capacity, $kJ/°C$
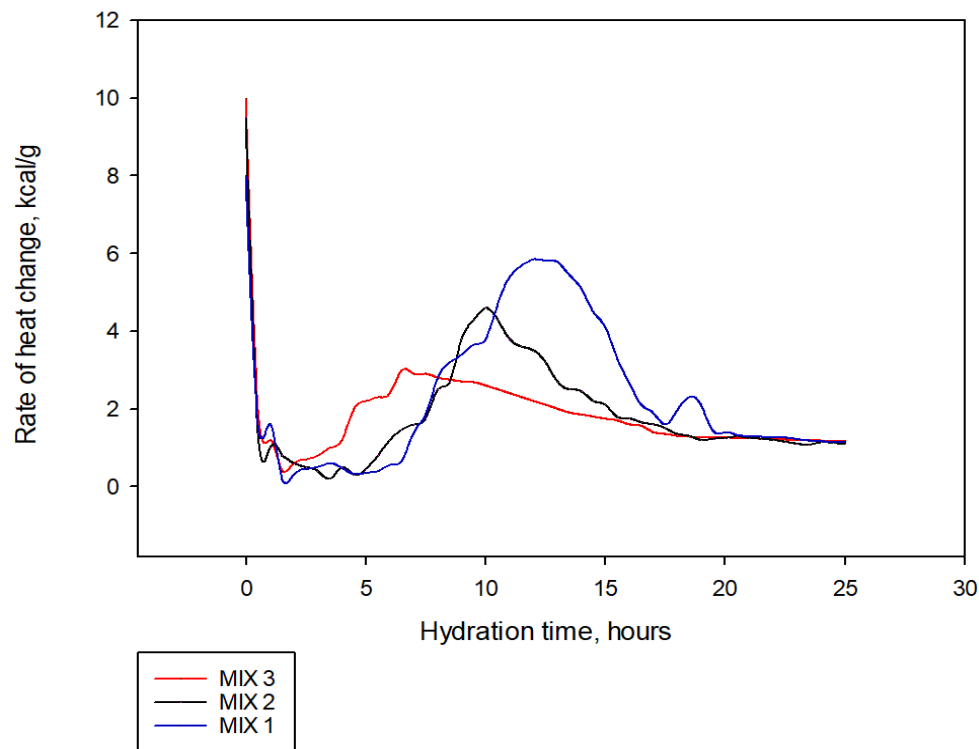
W_i= mass of the cement mix, g

t= final calorimeter temperature, °C

T= temperature of the cement mix, °C

R= corrected temperature, °C

The heat of hydration was calculated using equations 1-3 above. The outcomes of the calculations were illustrated as shown in Figure 4 below as the variation of heat during hydration of the three samples with hydration time.

Figure 4: Rate of Heat Change of Hydrating Cement.

Fly Ash

Fly ash as a filler material for massive structures is mainly used to alter the heat generated by cement during the hydration process. (Nili & Salehi, 2010) demonstrated that fly ash had the effect of decreasing the maximum peak temperature and the time sharpness of the temperature rise profiles. In this study, the effect of changing the concentration of fly ash on the cement mix is carried out. (Moghaddam & Vessalas, 2019) conducted an experiment that showed that the cumulative heat of hydration of blended cement paste decreased as the fly ash content in the blended cement paste was increased. The quality of fly ash also plays a pivotal role in changing the heat generated during hydration as was studied by (Karoriya & Gupta, 2016). Hence throughout this experiment, the quality of fly ash used was fly ash class C in accordance with the (ASTM 618, 2002) standards. Fly-Ash class C contains 20% calcium oxide (CaO) and was shown to have a greater cementitious characteristic as compared to class F fly ash (Ge, 2021).
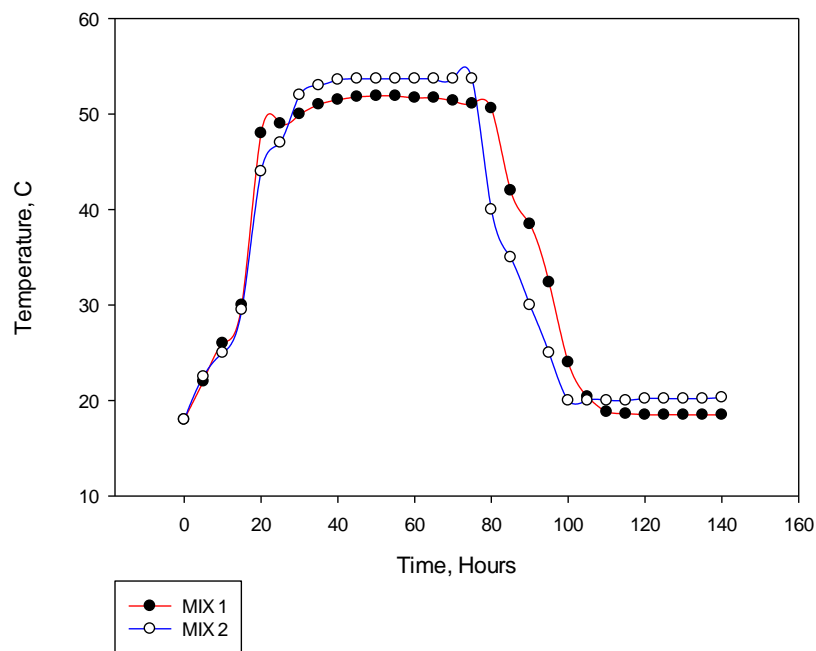
Experiment 2 Setup

The experiment seeks to establish the effect of varying fly ash concentration on temperature changes and rate of heat change

during the hydration process. Two samples of 100 grams of ordinary Portland cement are mixed with 50 % and 70% of fly ash by mass ratio as shown in Table 3 below. A water-cement ratio of 0.4 was used. The mixtures are then stirred at room temperature of $23 \pm 2°C$ for 10 minutes. The mixtures were then left to cure for 140 hours whilst temperatures were constantly recorded using a calorimeter. The results obtained from the temperature data logger are shown in Figure 5 below.
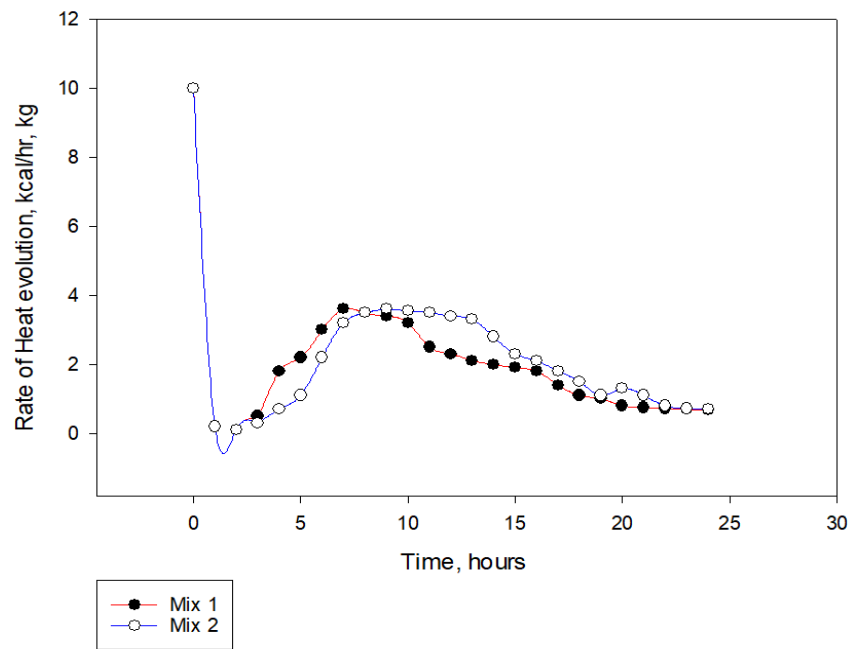
Table 3: Material Mix for Experiment 2

Material Mix	Cement Content, g	Fly Ash, g	Water, g
Mix 1	100	50	40
Mix 2	100	70	40

Figure 5: Temperature Changes on Cement Mix with Different Concentrations of Fly Ash.

Figure 6: Rate of Heat Change of Cement Mixes with Different Concentrations of Fly Ash.
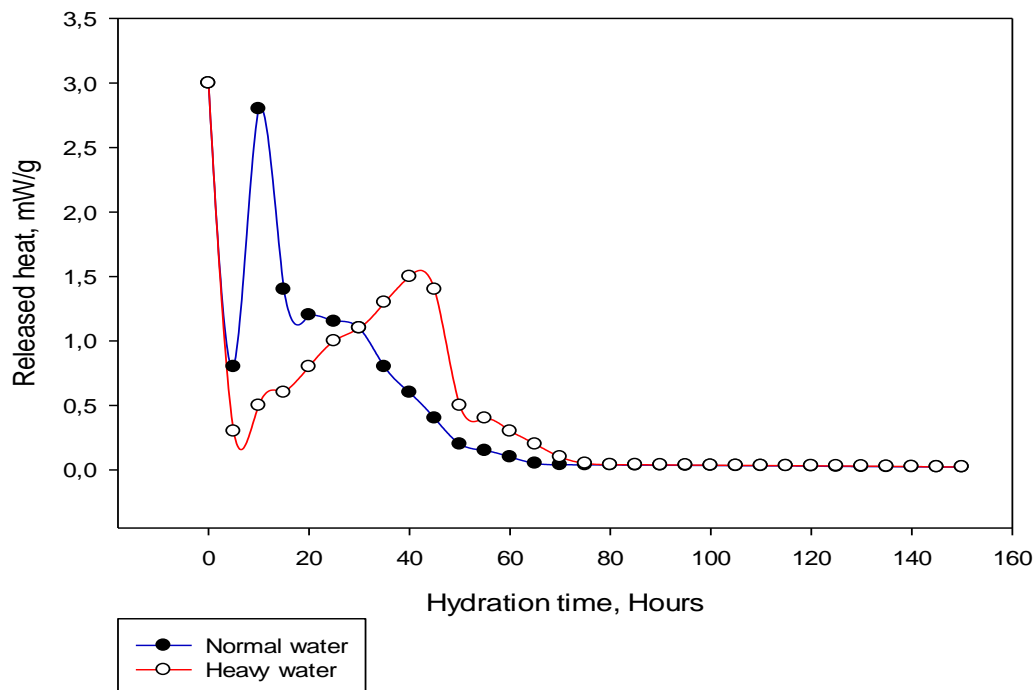
Heavy Water

Water acts as a solvent for cement as well as initiating the hydration process when mixed with cement. Water has a high polar character and tendency to form strong hydrogen bonds hence it being an excellent solvent. Heavy water is water that has the two hydrogen atoms substituted by a heavier isotope deuterium to give heavy water, D_2O, which has slightly weaker hydrogen bonds and has a higher ability to dissolve ions (Soper & Benmore, 2008). They showed that on the hydration heat of cement, heavy water has a retarding effect. The following experiment was conducted to determine the variation of heat released during hydration when two different types of water are used.

Experiment 3 Setup

Cement samples of 100 grams each are used in this experiment. The first sample is mixed with 40 grams of heavy water (D_2O), whilst the second sample is mixed with 40 grams of water (H_2O) as is shown in Table 4 below. The mixtures are stirred vigorously for 5 minutes at room temperature and left for 150 hours. An isothermal calorimeter set up as in Figure 3 was used to measure the amount of heat variations for the two mixtures.

Table 4: Material Mix for Experiment 3

Material Mix	Cement, g	Water, g	Heavy Water, g
Mix 1	100	0	40
Mix 2	100	40	0

Figure 7: Released Heat on Portland Cement using Water and Heavy Water.

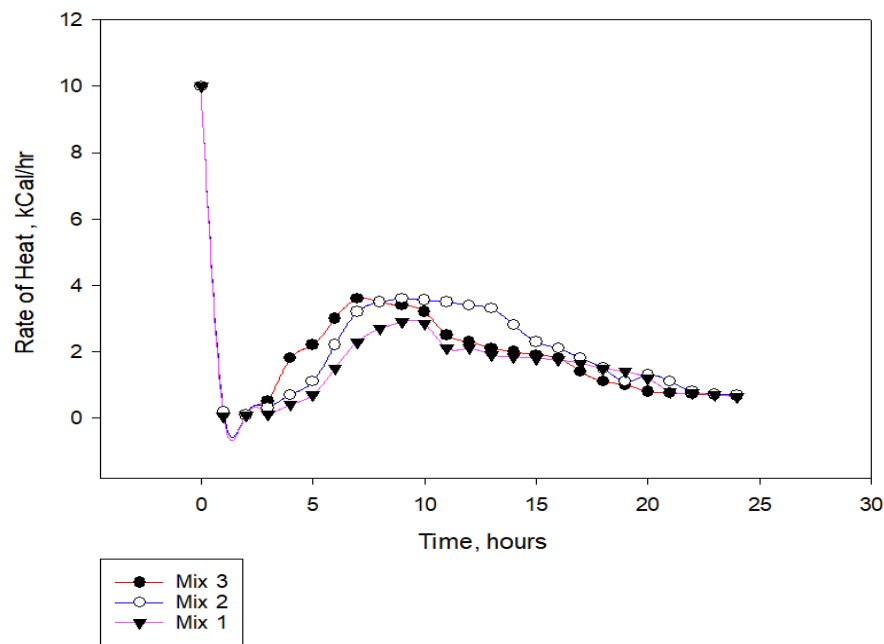**Combined Silica Fumes and Fly Ash**

The admixtures discussed in the sections above can be combined in order to effectively control the heat and temperature during the hydration process, as was demonstrated by (Langan, et al., 2002). When silica fumes and fly ash are added to the mixture, the concentration of Ca^{2+} ions reduced considerably as was demonstrated by (Lagundzija & Thiam, 2017). However, the hydration rate is heavily dependent on the water to cement ratio. Silica fumes accelerate the cement hydration at higher water to cement ratio and retards the cement hydration at low water to cement ratio. Fly ash on the other hand retards cement hydration more significantly at high water to cement ratio (Langan, et al., 2002). In this paper three mixtures of the different admixtures that have been mentioned in the experiments above. These mixtures were then analysed on their temperature and heat variation characteristics.

Experiment 4 Setup

The mixtures in this experiment were mixed as in the previous experiment.

Table 5: Material Mix for Experiment 4

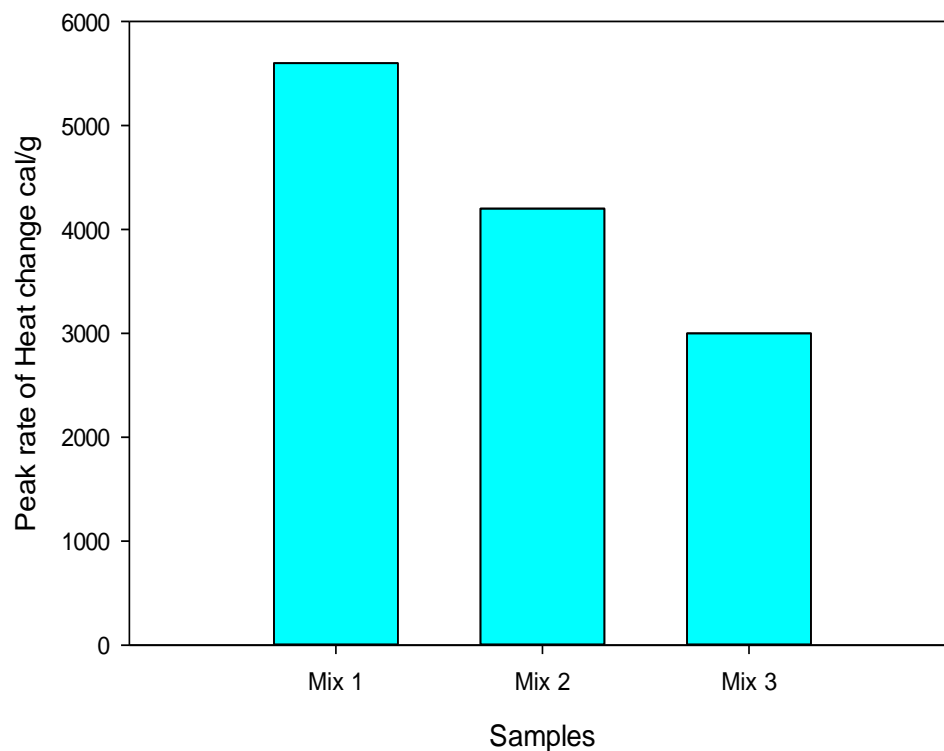| Material Mix	Silica Fumes	Fly Ash	Cement	Water
Mix 1	20	20	100	40
Mix 2	20	0	100	40
Mix 3	0	20	100	40

Figure 8: Released Heat on Portland Cement using a Mixed Combination of Silica Fumes and Fly Ash.

RESULTS AND DISCUSSIONS

Silica Fumes

The amount of heat measured during the experiment is a direct indication of hydration process since hydration is an exothermic reaction. Experimental 1 outcome showed that mix 1 had a steady and sharp increase in the rate of heat change followed by mix 2 and lastly mix 3. Figure 4 results indicate that mix 1, the control mix, without any silica fumes reached a peak of about 5 600 calories/g after 14 hours, mix 2 had a peak of 4 200 calories/ g and mix 3 had a peak of 3 000 calories/ g. The peak rate of heat change is as illustrated in Figure 9 below. The peak rate of heat change for sample mix 2 and mix 3 showed to be lower than that of mix 1. The rate of heat change is a measure of the hydration process; hence it can be concluded from Figure 9 that the hydration heat of the two samples containing silica fumes was reduced.
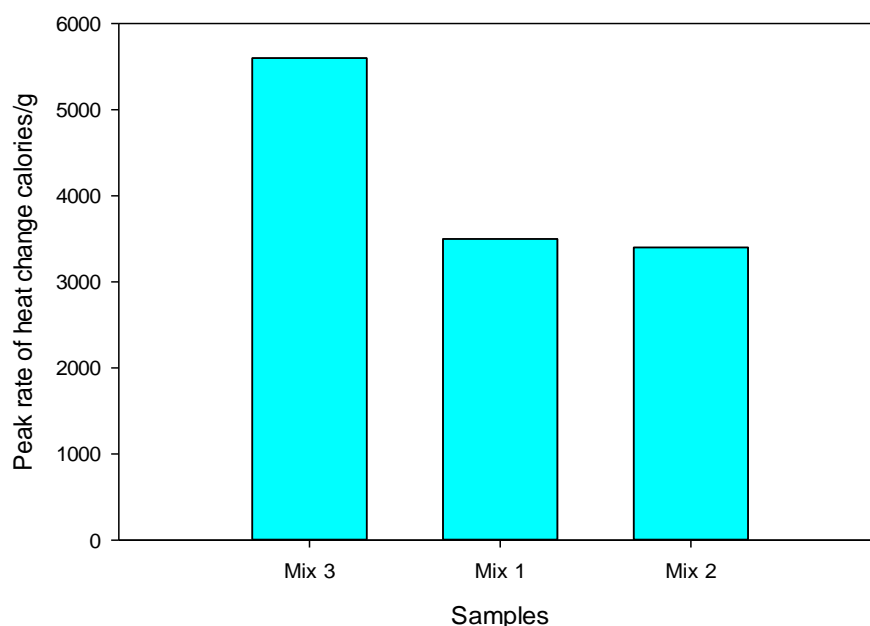
Figure 9: Peak of the Rate of Heat Change.

These findings are consistent with previous studies conducted by (Ribeiro, et al., 2020) which indicate that composites of cement paste with silica fumes exhibit a lower exothermic reaction. According to (Lagundzija & Thiam, 2017), the hydration process for samples of cement paste with silica fumes is reduced because the reaction between water and cement is exothermic, whereas the reaction between water and silica fumes is endothermic. Hence from the experimental results shown in Figure 4 and on Figure 9, the addition of silica fumes and increasing its concentration will reduce the exothermic reaction thereby reducing the hydration heat and temperature.
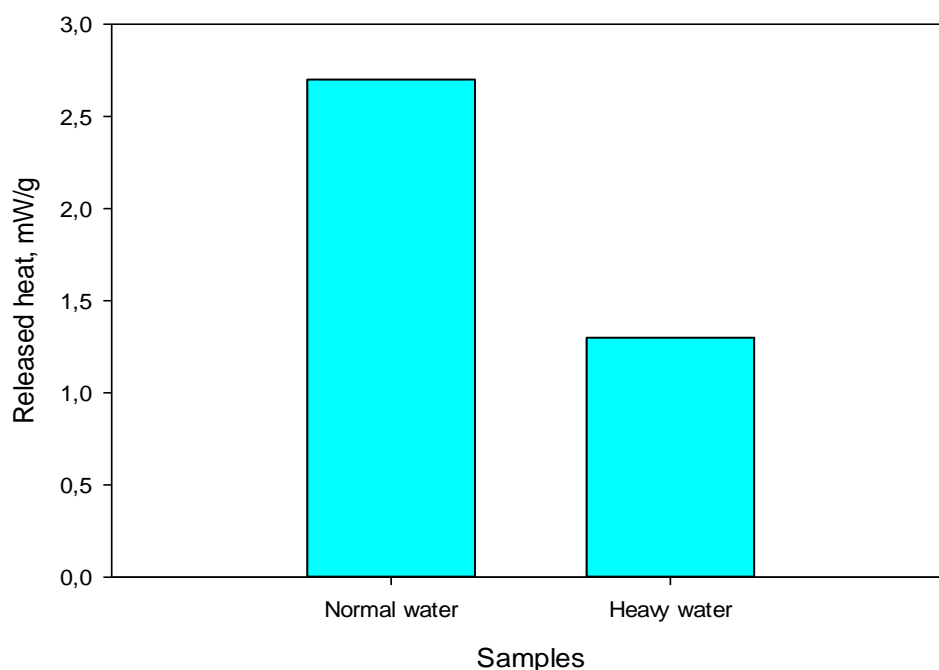
Fly Ash

Fly Ash has been shown to decrease the temperature and heat of hydration when a certain amount of the fly ash replaces cement (Ganjigatti, et al., 2015). Figure 5 shows that mix 2 with 70% fly ash had a lower rate of change of temperature for the first 25 hours. The hydration process of mix 2 lasted about 35 hours which is less than that of mix 1 of about 50 hours. The rate of heat evolution of mix 2 accelerated slower than that of mix 1. The peak rate of heat change shown in Figure 10 below shows that mix 1 and mix 2 which are samples with concentrations of fly ash had a lower peak relative to mix 3 which is a sample of only cement paste and water. However, from these experimental results, there is no conclusive evidence to indicate that a further increase in the concentration of the fly ash in cement paste will result in a further reduction of the hydration heat and temperature during the hydration process.

Figure 10: Peak Rate of Heat Change for Samples with Fly Ash.
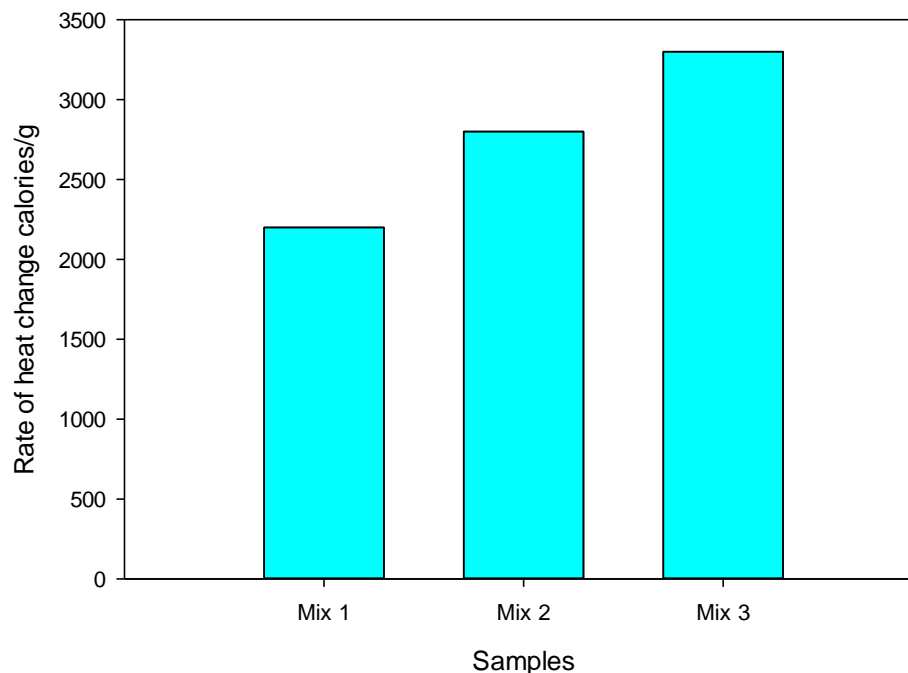
Heavy Water

The use of heavy water shown in Figure 7 indicates that the heavier deuterium isotope delays the hydration process. Heavy water mix achieved the second peak after 42 hours at 1.3mW/g compared to that of normal water after 18 hours at 2.7mW/g as is shown in Figure 11 below. The use of heavy water as a solvent for cement thus has the effect of reducing the heat and temperature produced by the cement water mixture.

Figure 11: Released Heat Comparison of Normal and Heavy Water During Hydration Process.

Combined Silica Fumes and Fly Ash

When fly ash and silica fumes are both used, the hydration is considerably delayed as shown on the outcome plot in Figure 8. Mix 1 sample which had silica fumes, fly ash and cement paste had a lower rate of heat change of 2 200 calories/g compared to mix 2 and mix 3 as shown in Figure 12 below. The increase of fly ash and silicone fumes reduces the concentration of calcium ions thereby reducing the concentration of the catalyst that reacts with silica fumes.

Figure 12: Rate of Heat Change for Combined Mixtures.

CONCLUSIONS

This paper focused on the methods that can be used to control the temperature and heat of hydration of cement. Three admixtures were studied on how they can be altered to control the hydration temperature and heat. Silica fumes as an admixture had a greater effect on altering the hydration process compared to fly ash as an admixture. By increasing the concentration of silica fumes mixed with cement paste from 10% to 20% resulted in a 21.4% decline in the peak rate of heat hydration whereas increasing the concentration of fly ash from 50% to 70% only resulted in a 1.7% decrease in the rate of change of the hydration heat. Combining silica fumes and fly ash as admixtures to cement paste in experiment 4 showed that when mixed in equal proportions of 20% the rate of heat change could be significantly reduced by 41%. However further increasing the concentration of fly ash as shown in experiment 2 did not show a linear negative relation with the rate of heat change.

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